Succinic acid semi-amides as anti-corrosive agents

The present invention relates to compositions comprising succinic acid semi-amides and to the use of those compositions in improving the use properties of lubricants, such as hydraulic or metal-working fluids, greases, gear oils or engine oils.

Additives that have to fulfil demanding tasks, such as a high load-bearing capacity, protection against wear and corrosion, and antioxidant action, are added to lubricants. In addition, the properties of lubricants should not be modified disadvantageously in the presence of contaminants. Often, in mineral oil industry enterprises, oils are admixed with commercially available additive concentrates or "packages". Contamination with water, calcium detergents and residues of other lubricants may occur during the mixing process. As a result, the action of the additive components with respect to corrosion is reduced, for example owing to formation of hydrolysis products and precipitation of poorly soluble calcium residues, the latter being especially disadvantageous because they block filter systems.

The present invention relates to the problem of producing lubricant compositions that provide improved protection against corrosion, combined with good compatibility with calcium ions.

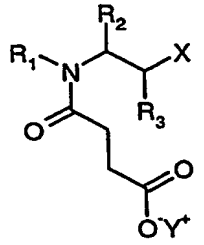
U.S. Patent Specification 4 462 918 discloses lubricant compositions providing protection against wear and corrosion, which comprise a component of the N-acyl-N-alkylaminosuccinic acid ester type (aspartic acid esters, aspartates).

U.S. Patent Specification 5 275 749 discloses lubricant compositions providing protection against wear and corrosion, which comprise a component of the N-acyl-N-alkoxyalkylamino-succinic acid ester type.

It has now been found, surprisingly, that succinic acid semi-amides, which are obtainable by reacting succinic acid anhydride with β-amino acid derivatives, improve the corrosion protection in lubricant compositions whilst at the same time the formation of precipitation products and residues is reduced.

The invention relates to compositions comprising

a) At least one compound of formula



(I),

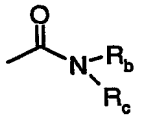
Wherein

R_1 is a substituent selected from the group consisting of C_1-C_{22}alkyl, C_2-C_{22}alkyl substituted by hydroxy, C_2-C_{22}alkyl interrupted by -C(=O)-, -O-C(=O)- or by -NR_a-C(=O)-, C_3-C_{22}alkyl interrupted by -O-, -S-, -NR_a-, -C(=O)-O- or by -C(=O)-NR_a-, wherein R_a denotes hydrogen or C_1-C_{22}alkyl, phenyl, benzyl, 1- or 2-phenylethyl, 2-phenoxyethyl, furfuryl, 1-naphthyl, 1-naphthylmethyl, cyclohexyl, cyclohexylmethyl, and isobornyl;

R_2 and R_3 are hydrogen, or one of R_2 and R_3 is hydrogen and the other is methyl; and

X is carboxy or carboxylate and Y^+ is a salt-forming cation suitable for lubricant compositions; or

X is derivatised carboxy selected from the group consisting of cyano, carboxy esterified by C_1-C_{22}alkyl, carboxy esterified by hydroxy-C_2-C_{22}alkyl, carboxy esterified by C_2-C_{22}alkyl interrupted by -C(=O)-, -C(=O)-O- or by -C(=O)-NR_a-, carboxy esterified by C_3-C_{22}alkyl interrupted by -O-, -S-, -NR_a-, -O-C(=O)- or by -NR_a-(C=O)-, wherein R_a denotes hydrogen or C_1-C_{22}alkyl, carboxy esterified by phenyl, benzyl, 1- or 2-phenylethyl, 2-phenoxyethyl, furfuryl, 1-naphthyl, 1-naphthylmethyl, cyclohexyl, cyclohexylmethyl, isobornyl, and carbamoyl of the partial formula



(A),

Wherein R_b and R_c are each independently of the other hydrogen, C_1-C_{22}alkyl or 2-hydroxyethyl, or R_b and R_c together are C_2-C_8alkylene, C_2-C_8alkenylene, C_2-C_8alkadienylene or C_2-C_8alkylene, C_2-C_8alkenylene or C_2-C_8alkadienylene interrupted by -O- or by -NR_a- with R_a being as defined; and

Y^+ is a hydrogen ion or is a salt-forming cation suitable for lubricant compositions; and

b) A base oil of lubricating viscosity.

The compounds (I) have excellent corrosion-inhibiting action combined with good compatibility with calcium ions, which may be present in lubricants especially as constituents of detergents.

A preferred embodiment relates to compositions comprising

a) At least one compound (I), wherein

R_1 is a substituent selected from the group consisting of C_1-C_{22}alkyl, C_2-C_{22}alkyl substituted by hydroxy, C_2-C_{22}alkyl interrupted by -C(=O)-, -O-C(=O)- or by -NR_a-C(=O)-, C_3-C_{22}alkyl interrupted by -O-, -S-, -NR_a-, -C(=O)-O- or by -C(=O)-NR_a-, wherein R_a denotes hydrogen or C_1-C_{22}alkyl, phenyl, benzyl, 1- or 2-phenylethyl, 2-phenoxyethyl, furfuryl, 1-naphthyl, 1-naphthylmethyl, cyclohexyl, cyclohexylmethyl and isobornyl;

R_2 and R_3 are hydrogen, or one of R_2 and R_3 is hydrogen and the other is methyl;

X is derivatised carboxy selected from the group consisting of cyano, carboxy esterified by C_1-C_{22}alkyl, carboxy esterified by hydroxy-C_2-C_{22}alkyl, carboxy esterified by C_2-C_{22}alkyl interrupted by -C(=O)-, -C(=O)-O- or by -C(=O)-NR_a-, carboxy esterified by C_3-C_{22}alkyl interrupted by -O-, -S-, -NR_a-, -O-C(=O)- or by -NR_a-(C=O)-, wherein R_a denotes hydrogen or C_1-C_{22}alkyl, carboxy esterified by phenyl, benzyl, 1- or 2-phenylethyl, 2-phenoxyethyl, furfuryl, 1-naphthyl, 1-naphthylmethyl, cyclohexyl, cyclohexylmethyl, isobornyl, and carbamoyl of the partial formula (A), wherein R_b and R_c are each independently of the other hydrogen, C_1-C_{22}alkyl or 2-hydroxyethyl, or R_b and R_c together are C_2-C_8alkylene, C_2-C_8alkenylene, C_2-C_8alkadienylene or C_2-C_8alkylene, C_2-C_8alkenylene or C_2-C_8alkadienylene interrupted by -O- or by -NR_a-, R_a being as defined; and

Y^+ is a hydrogen ion or is a salt-forming cation suitable for lubricant compositions; and

b) A base oil of lubricating viscosity.

An especially preferred embodiment corresponds to the following compositions comprising

a) At least one compound (I), wherein

R_1 is a substituent selected from the group consisting of C_1-C_{22}alkyl, C_2-C_{22}alkyl interrupted by -C(=O)- or by -O-C(=O)-, C_3-C_{22}alkyl interrupted by -O- , -S- or by -C(=O)-O-, phenyl and benzyl;

R_2 and R_3 are hydrogen;

X is derivatised carboxy selected from the group consisting of cyano, carboxy esterified by C_1-C_{22}alkyl, carboxy esterified by hydroxy-C_2-C_{22}alkyl, carboxy esterified by C_2-C_{22}alkyl interrupted by -C(=O)- or by -C(=O)-O-, carboxy esterified by C_3-C_{22}alkyl interrupted by -O-, -S- or by -O-C(=O)-, and carbamoyl of the partial formula (A) defined as heterocyclylcarbonyl; and

Y^+ is a hydrogen ion, ammonium, $(C_1$-C_4alkyl$)_{1-4}$ammonium or (2-hydroxyethyl$)_{1-4}$-ammonium; and

b) A base oil of lubricating viscosity.

A very especially preferred embodiment relates to compositions comprising

a) At least one compound (I), wherein

R_1 is a substituent selected from the group consisting of C_1-C_{22}alkyl, C_3-C_{22}alkyl interrupted by -O-, phenyl, and benzyl;

R_2 and R_3 are hydrogen;

X is derivatised carboxy selected from the group consisting of cyano, carboxy esterified by C_1-C_{22}alkyl, carboxy esterified by C_3-C_{22}alkyl interrupted by -O-, and carbamoyl of the partial formula (A) defined as piperidinocarbonyl, piperazinylcarbonyl or morpholinocarbonyl; and

Y^+ is a hydrogen ion, ammonium, $(C_1$-C_4alkyl$)_{1-4}$ammonium or (2-hydroxyethyl)$_{1-4}$-ammonium; and

b) A base oil of lubricating viscosity.

A likewise very especially preferred embodiment relates to compositions comprising

a) At least one compound (I), wherein

R_1 is a substituent selected from the group consisting of C_1-C_{18}alkyl, C_3-C_{18}alkyl interrupted by -O-, phenyl, and benzyl;

R_2 and R_3 are hydrogen;

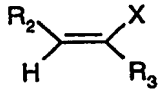
X is carboxy and Y is ammonium, $(C_1$-C_4alkyl$)_{1-4}$ammonium or (2-hydroxyethyl)$_{1-4}$-ammonium; or

X is carboxylate or derivatised carboxy selected from the group consisting of cyano, carboxy esterified by C_1-C_{18}alkyl, carboxy esterified by C_3-C_{18}alkyl interrupted by -O-, and morpholinocarbamoyl; and

Y^+ is hydrogen, ammonium, $(C_1$-C_4alkyl$)_{1-4}$ammonium or (2-hydroxyethyl)$_{1-4}$ammonium; and

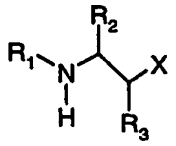
b) A base oil of lubricating viscosity.

The compounds (I) are prepared in a manner known *per se*, for example by addition of a primary amine R_1-NH_2 to an acrylic acid derivative:



(X = carboxy, carboxylate or derivatised carboxy)

(Addition of amines to α,β-unsaturated carbonyl compounds) to form a β-amino acid derivative:



Which is acylated with a reactive, functional derivative of succinic acid, for example succinic anhydride or succinic acid monochloride.

DE-A-2 054 649 describes the addition of primary amines to acrylic acid esters and the subsequent reaction with succinic anhydride. The compounds described therein can be used, *inter alia*, as textile adjuvants.

The terms and general designations used in the description of the present invention are preferably defined as follows:

Component a) Compounds (I)

R_1 and R_a defined as C_1-C_{22}alkyl include saturated, unbranched or, where possible, branched hydrocarbon groups, especially C_1-C_9alkyl, e.g. methyl, ethyl, isopropyl, n-butyl, isobutyl, tert-butyl, n-pentyl, neopentyl, isopentyl, n-hexyl, 2-ethylbutyl, 1-methylpentyl, 1,3-dimethylbutyl, n-heptyl, 3-heptyl, 1-methylhexyl, isoheptyl, n-octyl, 2-ethylhexyl, 1,1,3,3-tetramethylbutyl, 1-methylheptyl, n-nonyl or 1,1,3-trimethylhexyl, and also C_{10}-C_{22}alkyl, especially straight-chain C_{10}-C_{22}alkyl, e.g. n-decyl, n-dodecyl, n-tetradecyl, n-hexadecyl or n-octadecyl or branched C_{10}-C_{22}alkyl, e.g. 1,1,3-trimethylhexyl, 1-methylundecyl, 2-n-butyl-n-octyl, isotridecyl, 2-n-hexyl-n-decyl or 2-n-octyl-n-dodecyl, or higher homologues thereof.

R_1 defined as C_2-C_{22}alkyl substituted by hydroxy includes saturated, unbranched hydrocarbon groups having preferably from 2 to 9 carbon atoms, e.g. 2-hydroxyethyl or 2- or 3-hydroxypropyl.

R_1 defined as C_2-C_{22}alkyl interrupted by -C(=O)-, -O-C(=O)- or by -NR_a-C(=O)- includes unbranched or branched hydrocarbon groups preferably having from 2 to 9 carbon atoms, e.g. acetonyl, acetoxycarbonylmethyl, 2-acetoxycarbonylethyl, 2-tert-butoxycarbonylethyl or N,N-diethylcarbamoyl.

R_1 defined as C_3-C_{22}alkyl interrupted by -O-, -S-, -NR_a-, -C(=O)-O- or by -C(=O)-NR_a- includes unbranched or branched hydrocarbon groups preferably having from 3 to 18 carbon atoms, e.g. 2-methoxyethyl, 2- or 3-methoxypropyl, 2-, 3- or 4-methoxybutyl, 2-ethoxyethyl, 2- or 3-ethoxypropyl, 2-, 3- or 4-ethoxybutyl, 2-n-propoxyethyl, 2- or 3-n-propoxypropyl, 2-, 3- or 4-n-propoxybutyl, 2-isopropoxyethyl, 2- or 3-isopropoxypropyl, 2-, 3- or 4-isopropoxybutyl, 2-n-butoxyethyl, 2- or 3-n-butoxypropyl, 2-, 3- or 4-n-butoxybutyl, 2-tert-butoxyethyl, 2- or 3-tert-butoxypropyl, 2-, 3- or 4-tert-butoxybutyl, 2-methylthioethyl, 2- or 3-methylthiopropyl, 2-

ethylthioethyl, 2-dimethylaminoethyl, 2- or 3-dimethylaminopropyl, 2-diethylaminoethyl, 2- or 3-diethylaminopropyl, 2-acetoxyethyl or 2-(N-acetoxy-N-methyl)-ethyl.

In compounds (I), R_2 and R_3 are hydrogen, or one of R_2 and R_3 is hydrogen and the other is methyl. Preferably, R_2 and R_3 are hydrogen.

When X in compounds (I) is carboxy or carboxylate [-C(=O)-O$^-$], Y^+ is a salt-forming cation suitable for lubricant compositions, e.g. ammonium, tetramethylammonium, tetraethylammonium or 2-hydroxyethyltrimethylammonium. There is present only one carboxy group in substantially undissociated form [-C(=O)-OH] or in salt form [-C(=O)-O$^-$], e.g. with ammonium ions, e.g. ammonium, tetramethylammonium, tetraethylammonium or 2-hydroxyethyltrimethylammonium.

In a preferred embodiment, X is derivatised carboxy or unsubstituted or substituted carbamoyl as defined hereinbelow. Y^+ is then a hydrogen ion or a salt-forming cation suitable for lubricant compositions.

X defined as carboxy esterified by C_1-C_{22}alkyl is, for example, a carboxy group which is esterified by the C_1-C_{22}alkyl groups defined hereinabove for R_1, e.g. saturated, unbranched or, where possible, branched hydrocarbon groups, e.g. methyl, ethyl, isopropyl, n-butyl, isobutyl, tert-butyl, n-pentyl, neopentyl, isopentyl, n-hexyl, 2-ethylbutyl, 1-methylpentyl, 1,3-dimethylbutyl, n-heptyl, 3-heptyl, 1-methylhexyl, isoheptyl, n-octyl, 2-ethylhexyl, 1,1,3,3-tetramethylbutyl, 1-methylheptyl, n-nonyl or 1,1,3-trimethylhexyl, and also by C_{10}-C_{22}alkyl, especially straight-chain C_{10}-C_{22}alkyl, e. g. n-decyl, n-dodecyl, n-tetradecyl, n-hexadecyl or n-octadecyl.

X defined as carboxy esterified by hydroxy-C_2-C_{22}alkyl is, for example, a carboxy group that is esterified by hydroxy-C2-C22alkyl defined hereinabove for R_1, e.g. 2-hydroxyethoxycarbonyl or 2- or 3-hydroxypropoxycarbonyl.

X defined as carboxy esterified by C_2-C_{22}alkyl interrupted by -C(=O)-, -C(=O)-O- or by -C(=O)-NR_a- is, for example, a carboxy group esterified by C_2-C_{22}alkyl, defined hereinabove for R_1, interrupted by -C(=O)-, -C(=O)-O- or -C(=O)-NR_a-, e.g. acetonyloxycarbonyl, acetoxycarbonylmethoxycarbonyl or 2-acetoxycarbonylethoxycarbonyl.

X defined as carboxy esterified by C_3-C_{22}alkyl interrupted by -O-, -S-, -NR_a-, -O-C(=O)- or by -NR_a-(C=O)- is, for example, a carboxy group esterified by C_3-C_{22}alkyl (defined hereinabove for R_1) interrupted by -O-, -S-, -NR_a-, -O-C(=O)- or by -NR_a-(C=O)-, e.g. 2-methoxyethoxycarbonyl, 2- or 3-methoxypropoxycarbonyl, 2-, 3- or 4-methoxybutoxycarbonyl, 2-ethoxyethoxycarbonyl, 2- or 3-ethoxypropoxycarbonyl, 2-, 3- or 4-ethoxybutoxycarbonyl, 2-methylthioethoxycarbonyl, 2- or 3-methylthiopropoxycarbonyl, 2-ethylthioethoxycarbonyl,

2-dimethylaminoethoxycarbonyl, 2- or 3-dimethylaminopropoxycarbonyl, 2-diethylaminoethoxycarbonyl, 2- or 3-diethylaminopropoxycarbonyl, 2-acetoxyethoxycarbonyl or 2-(N-acetoxy-N-methyl)-ethoxycarbonyl.

X defined as carbamoyl of the partial formula A is, for example, carbamoyl, dimethyl- or diethyl-carbamoyl. In addition, R_b and R_c may be linked to one another by C_2-C_8alkylene, e.g. 1,4-n-butylene or 1,5-n-pentylene, by C_2-C_8alkenylene, e.g. 2-butenylene, or by C_2-C_8alkadienylene, e.g. 1,3-butadienylene and, together with –N<, form a heterocycle which may, in turn, contain further hetero atoms, e.g. N or O. In that case, the partial formula A corresponds to a heterocyclylcarbonyl substituent, e.g. piperidinocarbonyl, piperazinylcarbonyl or morpholinocarbonyl.

In the compounds (I) described hereinabove, the sum of the carbon atoms present in R_1 and X is preferably greater than ten.

The definition used for Y^+, "a salt-forming cation suitable for lubricant compositions", includes those cations of salt-formers that together with the carboxylate group form metal salts suitable for lubricant compositions, e.g. alkali metal, alkaline earth metal, zinc (Zn^{2+}) or copper (Cu^{2+}) salts, e.g. sodium, potassium, calcium, zinc^{2+} or Cu^{2+} ions.

In a preferred embodiment of the invention, the definition used for Y^+, "a salt-forming cation suitable for lubricant compositions", is understood to mean those cations of salt-formers that together with the carboxylate group form suitable, non-metallic salts, e.g. ammonium, (C_1-C_{22}alkyl)$_{1-4}$ammonium or (2-hydroxyethyl)$_{1-4}$ammonium, e.g. tetramethylammonium, tetraethylammonium or 2-hydroxyethyltrimethylammonium.

The compounds (I) are readily soluble in oils and can be admixed with a base oil of lubricating viscosity, e.g. a lubricant, in a manner known *per se*.

Component b) Base oil

The definition "base oil of lubricating viscosity" includes, for example, lubricants that can be used for hydraulic or metal-working fluids, greases, gear oils or engine oils.

Suitable lubricants are based, for example, on mineral or synthetic oils or mixtures thereof. The person skilled in the art will be familiar with the lubricants, which are described in the relevant technical literature, for example in *Chemistry and Technology of Lubricants; Mortier, R.M. and Orszulik, S.T. (Editors); 1992 Blackie and Son Ltd. for GB, VCH-Publishers N.Y. for U.S., ISBN 0-216-92921-0,* see pages 208 ff and 269 ff; in *Kirk-Othmer Encyclopedia of Chemical Technology, Fourth Edition 1969, J. Wiley & Sons, New York, Vol. 13,* page 533 ff *(Hydraulic Fluids); Performance Testing of Hydraulic Fluids; R. Tourret and E.P. Wright, Hyden & Son Ltd. GB, on behalf of The Institute of Petroleum London, ISBN 0 85501 317 6;*

Ullmann's Encyclopaedia of Ind. Chem., Fifth Completely Revised Edition, Verlag Chemie, DE-Weinheim, VCH-Publishers for U.S., Vol. A 15, page 423 ff *(Lubricants), Vol. A 13,* page 165 ff *(Hydraulic Fluids).*

The lubricants are especially oils and greases, for example based on mineral oil or vegetable and animal oils, greases, tallow and wax or mixtures thereof. Vegetable and animal oils, greases, tallow and wax are, for example, palm-kernel oil, palm oil, olive oil, rapeseed oil, rape oil, linseed oil, soybean oil, cottonseed oil, sunflower oil, coconut oil, maize oil, castor oil, tree nut oil and mixtures thereof, fish oils, and their chemically modified forms, for example epoxidised and sulphoxidised forms, or forms produced by genetic engineering, for example soybean oil produced by genetic engineering.

Examples of synthetic lubricants include lubricants based on aliphatic or aromatic carboxylic acid esters, polymeric esters, polyalkylene oxides, phosphoric acid esters, poly-α-olefins or silicones, diesters of a divalent acid with a monohydric alcohol, such as, for example, dioctyl sebacate or dinonyl adipate, triesters of trimethylolpropane with a monovalent acid or with a mixture of such acids, such as, for example, trimethylolpropane tripelargonate, trimethylolpropane tricaprylate or mixtures thereof, tetraesters of pentaerythritol with a monovalent acid or with a mixture of such acids, such as, for example, pentaerythritol tetracaprylate, or complex esters of monovalent and divalent acids with polyhydric alcohols, for example a complex ester of trimethylolpropane with caprylic and sebacic acid, or a mixture thereof. Apart from mineral oils there are especially suitable, for example, poly-α-olefins, ester-based lubricants, phosphates, glycols, polyglycols and polyalkylene glycols, and also mixtures thereof with water.

An organic or inorganic thickener can also be added to the mentioned lubricants or mixtures thereof (base grease). Metal-working fluids and hydraulic fluids can be prepared on the basis of the same substances as described hereinabove for the lubricants. Such fluids are frequently also emulsions of such substances in water or other liquids.

The compositions advantageously contain from 0.005 to 10.0 % by weight, preferably from 0.01 to 5.0 % by weight, especially from 0.01 to 0.9 % by weight, of compounds (I).

The compositions are used, for example, in hydraulic or metal-working fluids, greases, gear oils, or oils for engines of the Otto, diesel, two-stroke, Wankel or orbital type.

The mentioned lubricants may additionally comprise other additives that are added in order to improve their basic properties still further; such additives include: antioxidants, metal deactivators, rust inhibitors, viscosity index improvers, pour-point depressants, dispersants, detergents, extreme-pressure additives, anti-wear additives and friction reducers. Such addi-

tives are added in the customary amounts in each case, in the range of about from 0.01 to 10.0 % by weight of each. The following are examples of further additives:

Phenolic antioxidants

1. Alkylated monophenols: 2,6-di-tert-butyl-4-methylphenol, 2-butyl-4,6-dimethylphenol, 2,6-di-tert-butyl-4-ethylphenol, 2,6-di-tert-butyl-4-n-butylphenol, 2,6-di-tert-butyl-4-isobutylphenol, 2,6-dicyclopentyl-4-methylphenol, 2-(α-methylcyclohexyl)-4,6-dimethylphenol, 2,6-dioctadecyl-4-methylphenol, 2,4,6-tricyclohexylphenol, 2,6-di-tert-butyl-4-methoxymethylphenol, linear nonylphenols or nonylphenols branched in the side-chain, e.g. 2,6-dinonyl-4-methylphenol, 2,4-dimethyl-6-(1'-methylundec-1'-yl)-phenol, 2,4-dimethyl-6-(1'-methylheptadec-1'-yl)-phenol, 2,4-dimethyl-6-(1'-methyltridec-1'-yl)-phenol and mixtures thereof.

2. Alkylthiomethylphenols: 2,4-dioctylthiomethyl-6-tert-butylphenol, 2,4-dioctylthiomethyl-6-methylphenol, 2,4-dioctylthiomethyl-6-ethylphenol, 2,6-didodecylthiomethyl-4-nonylphenol.

3. Hydroquinones and alkylated hydroquinones: 2,6-di-tert-butyl-4-methoxyphenol, 2,5-di-tert-butylhydroquinone, 2,5-di-tert-amylhydroquinone, 2,6-diphenyl-4-octadecyloxyphenol, 2,6-di-tert-butylhydroquinone, 2,5-di-tert-butyl-4-hydroxyanisole, 3,5-di-tert-butyl-4-hydroxyanisole, 3,5-di-tert-butyl-4-hydroxyphenyl stearate, bis(3,5-di-tert-butyl-4-hydroxyphenyl) adipate.

4. Tocopherols: α-, β-, γ- or δ-tocopherols and mixtures thereof (vitamin E).

5. Hydroxylated thiodiphenyl ethers: 2,2'-thiobis(6-tert-butyl-4-methylphenol), 2,2'-thiobis(4-octylphenol), 4,4'-thiobis(6-tert-butyl-3-methylphenol), 4,4'-thiobis(6-tert-butyl-2-methylphenol), 4,4'-thiobis(3,6-di-sec-amylphenol), 4,4'-bis(2,6-dimethyl-4-hydroxyphenyl) disulphide.

6. Alkylidene bisphenols: 2,2'-methylenebis(6-tert-butyl-4-methylphenol), 2,2'-methylenebis(6-tert-butyl-4-ethylphenol), 2,2'-methylenebis[4-methyl-6-(α-methylcyclohexyl)phenol], 2,2'-methylenebis(4-methyl-6-cyclohexylphenol), 2,2'-methylenebis(6-nonyl-4-methylphenol), 2,2'-methylenebis(4,6-di-tert-butylphenol), 2,2'-ethylidenebis(4,6-di-tert-butylphenol), 2,2'-ethylidenebis(6-tert-butyl-4-isobutylphenol), 2,2'-methylenebis[6-(α-methylbenzyl)-4-nonylphenol], 2,2'-methylenebis[6-(α,α-dimethylbenzyl)-4-nonylphenol], 4,4'-methylenebis(2,6-di-tert-butylphenol), 4,4'-methylenebis(6-tert-butyl-2-methylphenol), 1,1-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)butane, 2,6-bis(3-tert-butyl-5-methyl-2-hydroxybenzyl)-4-methylphenol, 1,1,3-tris(5-tert-butyl-4-hydroxy-2-methylphenyl)butane, 1,1-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)-3-n-dodecylmercaptobutane, ethylene glycol bis[3,3-bis(3'-tert-butyl-4'-hydroxyphenyl)butyrate], bis(3-tert-butyl-4-hydroxy-5-methylphenyl)dicyclopentadiene, bis[2-(3'-tert-butyl-2'-hydroxy-5'-methylbenzyl)-6-tert-butyl-4-

methylphenyl] terephthalate, 1,1-bis(3,5-dimethyl-2-hydroxyphenyl)butane, 2,2-bis(3,5-di-tert-butyl-4-hydroxyphenyl)propane, 2,2-bis(5-tert-butyl-4-hydroxy-2-methylphenyl)-4-n-dodecylmercaptobutane, 1,1,5,5-tetra(5-tert-butyl-4-hydroxy-2-methylphenyl)pentane.

7. O-, N- and S-benzyl compounds: 3,5,3',5'-tetra-tert-butyl-4,4'-dihydroxydibenzyl ether, octadecyl-4-hydroxy-3,5-dimethylbenzyl mercaptoacetate, tridecyl-4-hydroxy-3,5-di-tert-butylbenzyl mercaptoacetate, tris(3,5-di-tert-butyl-4-hydroxybenzyl)amine, bis(4-tert-butyl-3-hydroxy-2,6-dimethylbenzyl) dithioterephthalate, bis(3,5-di-tert-butyl-4-hydroxybenzyl) sulphide, isooctyl-3,5-di-tert-butyl-4-hydroxybenzyl mercaptoacetate.

8. Hydroxybenzylated malonates: dioctadecyl 2,2-bis(3,5-di-tert-butyl-2-hydroxybenzyl)malonate, dioctadecyl 2-(3-tert-butyl-4-hydroxy-5-methylbenzyl)malonate, didodecylmercaptoethyl 2,2-bis(3,5-di-tert-butyl-4-hydroxybenzyl)malonate, di-[4-(1,1,3,3-tetramethylbutyl)phenyl] 2,2-bis(3,5-di-tert-butyl-4-hydroxybenzyl)malonate.

9. Hydroxybenzyl aromatic compounds: 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-2,4,6-trimethylbenzene, 1,4-bis(3,5-di-tert-butyl-4-hydroxybenzyl)-2,3,5,6-tetramethylbenzene, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxybenzyl)phenol.

10. Triazine compounds: 2,4-bisoctylmercapto-6-(3,5-di-tert-butyl-4-hydroxyanilino)-1,3,5-triazine, 2-octylmercapto-4,6-bis(3,5-di-tert-butyl-4-hydroxyanilino)-1,3,5-triazine, 2-octylmercapto-4,6-bis(3,5-di-tert-butyl-4-hydroxyphenoxy)-1,3,5-triazine, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxyphenoxy)-1,2,3-triazine, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl) isocyanurate, 1,3,5-tris(4-tert-butyl-3-hydroxy-2,6-dimethylbenzyl) isocyanurate, 2,4,6-tris(3,5-di-tert-butyl-4-hydroxyphenylethyl)-1,3,5-triazine, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hexahydro-1,3,5-triazine, 1,3,5-tris(3,5-dicyclohexyl-4-hydroxybenzyl) isocyanurate.

11. Acylaminophenols: 4-hydroxylauric acid anilide, 4-hydroxystearic acid anilide, N-(3,5-di-tert-butyl-4-hydroxyphenyl)carbamic acid octyl ester.

12. Esters of beta-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid with mono- or poly-hydric alcohols, for example with methanol, ethanol, n-octanol, isooctanol, octadecanol, 1,6-hexanediol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl) isocyanurate, N,N'-bis(hydroxyethyl)oxalic acid diamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.

13. Esters of beta-(5-tert-butyl-4-hydroxy-3-methylphenyl)propionic acid (with mono- or poly-hydric alcohols): with methanol, ethanol, n-octanol, isooctanol, octadecanol, 1,6-hexane-

diol, 1,9-nonanediol, ethylene glycol, 1,2-propanediol, neopentyl glycol, thiodiethylene glycol, diethylene glycol, triethylene glycol, pentaerythritol, tris(hydroxyethyl) isocyanurate, N,N'-bis(hydroxyethyl)oxalic acid diamide, 3-thiaundecanol, 3-thiapentadecanol, trimethylhexanediol, trimethylolpropane, 4-hydroxymethyl-1-phospha-2,6,7-trioxabicyclo[2.2.2]octane.

14. Esters of beta-(3,5-dicyclohexyl-4-hydroxyphenyl)propionic acid with mono- or poly-hydric alcohols, e.g. with the alcohols mentioned under 13..

15. Esters of 3,5-di-tert-butyl-4-hydroxyphenylacetic acid with mono- or poly-hydric alcohols, e.g. with the alcohols mentioned under 13..

16. Amides of beta-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid, for example N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hexamethylenediamine, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)trimethylenediamine, N,N'-bis(3,5-di-tert-butyl-4-hydroxyphenylpropionyl)hydrazine.

Amine-type antioxidants

N,N'-Di-isopropyl-p-phenylenediamine, N,N'-di-sec-butyl-p-phenylenediamine, N,N'-bis(1,4-dimethylpentyl)-p-phenylenediamine, N,N'-bis(1-ethyl-3-methylpentyl)-p-phenylenediamine, N,N'-bis(1-methylheptyl)-p-phenylenediamine, N,N'-dicyclohexyl-p-phenylenediamine, N,N'-diphenyl-p-phenylenediamine, N,N'-di(2-naphthyl)-p-phenylenediamine, N-isopropyl-N'-phenyl-p-phenylenediamine, N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine, N-(1-methylheptyl)-N'-phenyl-p-phenylenediamine, N-cyclohexyl-N'-phenyl-p-phenylenediamine, 4-(p-toluenesulphonamido)-diphenylamine, N,N'-dimethyl-N,N'-di-sec-butyl-p-phenylenediamine, diphenylamine, N-allyldiphenylamine, 4-isopropoxydiphenylamine, N-phenyl-1-naphthylamine, N-(4-tert-octylphenyl)-1-naphthylamine, N-phenyl-2-naphthylamine, octylated diphenylamine, for example p,p'-di-tert-octyldiphenylamine, 4-n-butylaminophenol, 4-butyrylaminophenol, 4-nonanoylaminophenol, 4-dodecanoylaminophenol, 4-octadecanoylaminophenol, di(4-methoxyphenyl)amine, 2,6-di-tert-butyl-4-dimethylaminomethylphenol, 2,4'-diaminodiphenylmethane, 4,4'-diaminodiphenylmethane, N,N,N',N'-tetramethyl-4,4'-diaminodiphenylmethane, 1,2-di[(2-methylphenyl)amino]ethane, 1,2-di(phenylamino)propane, (o-tolyl)-biguanide, di[4-(1',3'-dimethylbutyl)phenyl]amine, tert-octylated N-phenyl-1-naphthylamine, mixture of mono- and di-alkylated tert-butyl-/tert-octyl-diphenylamines, mixture of mono- and di-alkylated nonyldiphenylamines, mixture of mono- and di-alkylated dodecyldiphenylamines, mixture of mono- and di-alkylated isopropyl-/isohexyl-diphenylamines, mixtures of mono- and di-alkylated tert-butyldiphenylamines, 2,3-dihydro-3,3-dimethyl-4H-1,4-benzothiazine, phenothiazine, mixture of mono- and di-alkylated tert-butyl-/tert-octyl-phenothiazines, mixture of mono- and di-alkylated tert-octylphenothiazines, N-allylphenothiazine, N,N,N',N'-tetraphenyl-

1,4-diaminobut-2-ene, N,N-bis(2,2,6,6-tetramethyl-piperidin-4-yl-hexamethylenediamine, bis(2,2,6,6-tetramethylpiperidin-4-yl) sebacate, 2,2,6,6-tetramethypiperidin-4-one, 2,2,6,6-tetramethylpiperidin-4-ol.

Further antioxidants

Ascorbic acid (vitamin C), aliphatic or aromatic phosphites, esters of thiodipropionic acid or thiodiacetic acid, or salts of dithiocarbamic or dithiophosphoric acid, 2,2,12,12-tetramethyl-5,9-dihydroxy-3,7,11-trithiatridecane and 2,2,15,15-tetramethyl-5,12-dihydroxy-3,7,10,14-tetrathiahexadecane.

Metal deactivators, for example for copper

1. Benzotriazoles and derivatives thereof: 2-mercaptobenzotriazole, 2,5-dimercaptobenzotriazole, 4- or 5-alkylbenzotriazoles (e.g. tolutriazole) and derivatives thereof, 4,5,6,7-tetrahydrobenzotriazole, 5,5'-methylene-bisbenzotriazole; Mannich bases of benzotriazole or tolutriazole, such as 1-[di(2-ethylhexyl)aminomethyl]tolutriazole and 1-[di(2-ethylhexyl)-aminomethyl]benzotriazole; alkoxyalkylbenzotriazoles, such as 1-(nonyloxymethyl)benzotriazole, 1-(1-butoxyethyl)benzotriazole and 1-(1-cyclohexyloxybutyl)tolutriazole.

2. 1,2,4-Triazoles and derivatives thereof: 3-alkyl- (or aryl-)1,2,4-triazoles, Mannich bases of 1,2,4-triazoles, such as 1-[di(2-ethylhexyl)aminomethyl]-1,2,4-triazole; alkoxyalkyl-1,2,4-triazoles, such as 1-(1-butoxyethyl)-1,2,4-triazole; acylated 3-amino-1,2,4-triazoles.

3. Imidazole derivatives: 4,4'-methylenebis(2-undecyl-5-methylimidazole); bis[(N-methyl)-imidazol-2-yl]carbinol-octyl ether.

4. Sulphur-containing heterocyclic compounds: 2-mercaptobenzothiazole, 2,5-dimercapto-1,3,4-thiadiazole, 2,5-dimercaptobenzothiadiazole and derivatives thereof; 3,5-bis[di(2-ethylhexyl)aminomethyl]-1,3,4-thiadiazolin-2-one.

5. Amino compounds: salicylidene-propylenediamine, salicylaminoguanidine and salts thereof.

Rust inhibitors

1. Organic acids, their esters, metal salts, amine salts and anhydrides: alkyl- and alkenyl-succinic acids and their partial esters with alcohols, diols or hydroxycarboxylic acids, partial amides of alkyl- and alkenyl-succinic acids, 4-nonylphenoxyacetic acid, alkoxy- and alkoxyethoxy-carboxylic acids, such as dodecyloxyacetic acid, dodecyloxy(ethoxy)acetic acid and amine salts thereof, and also N-oleoyl-sarcosine, sorbitan monooleate, lead naphthenate, alkenylsuccinic acid anhydrides, e.g. dodecenylsuccinic acid anhydride, 2-(2-carboxyethyl)-1-dodecyl-3-methylglycerol and salts thereof, especially sodium and triethanolamine salts thereof.

2. Nitrogen-containing compounds:

2.1. Tertiary aliphatic or cycloaliphatic amines and amine salts of organic and inorganic acids, e.g. oil-soluble alkylammonium carboxylates, and also 1-[N,N-bis(2-hydroxyethyl)amino]-3-(4-nonylphenoxy)propan-2-ol.

2.2. Heterocyclic compounds, e.g. substituted imidazolines and oxazolines, e.g. 2-heptadecenyl-1-(2-hydroxyethyl)-imidazoline.

3. Sulphur-containing compounds: barium dinonylnaphthalenesulphonates, calcium petroleum sulphonates, alkylthio-substituted aliphatic carboxylic acids, esters of aliphatic 2-sulphocarboxylic acids and salts thereof.

Extreme-pressure and anti-wear additives

Sulphur-containing and halogen-containing compounds, for example, chlorinated paraffins, sulphurated olefins or vegetable oils (soybean/rapeseed oil), alkyl- or aryl-di- or -tri-sulphides, benzotriazoles or derivatives thereof such as bis(2-ethylhexyl)aminomethyltolutriazole, dithiocarbamates such as methylene-bis-dibutyldithiocarbamate, derivatives of 2-mercaptobenzothiazole such as 1-[N,N-bis(2-ethylhexyl)aminomethyl]-2-mercapto-1H-1,3-benzothiazole, derivatives of 2,5-dimercapto-1,3,4-thiadiazole such as 2,5-bis(tert-nonyldithio)-1,3,4-thiadiazole.

Friction coefficient reducing agents

Lard oil, oleic acid, tallow, rapeseed oil, sulphurated fats, amines. Further examples are given in *EP-A-0 565 487*.

Further additives

1. Viscosity index improvers: polyacrylates, polymethacrylates, vinylpyrrolidone/methacrylate copolymers, polyvinylpyrrolidones, polybutenes, olefin copolymers, styrene/acrylate copolymers, polyethers.

2. Pour-point depressants: poly(meth)acrylates, ethylene/vinyl acetate copolymer, alkylpolystyrenes, fumarate copolymers, alkylated naphthalene derivatives.

3. Dispersants/surfactants: polybutenylsuccinic acid amides or imides, polybutenylphosphonic acid derivatives, basic magnesium, calcium and barium sulphonates and phenolates.

Special additives

For use in water/oil metal-working fluids and hydraulic fluids

1. Emulsifiers: petroleum sulphonates, amines, such as polyoxyethylated fatty amines, nonionic surface-active substances;

2. Buffers: alkanolamines;

3. Biocides: triazines, thiazolinones, tris-nitromethane, morpholine, sodium pyridenethiol;

4. Processing speed improvers: calcium and barium sulphonates.

The mentioned ingredients are added to the composition using customary mixing methods in a manner known *per se*. It is also possible, using the compounds (I) or mixtures thereof and, optionally, further additives, to prepare a concentrate or so-called additive package, which is diluted, as required for use, to a concentration for use in the lubricant in question. The composition of the components in the concentrate is such that the concentrate is liquid at room temperature without further addition of component b) or a solvent.

The present invention relates also to a concentrate comprising a) at least one compound (I) wherein R_1, R_2, R_3, X and Y are as defined, optionally further additives, and b) a base oil of lubricating viscosity.

The invention relates also to a method of improving the use properties of lubricants, which comprises adding to the lubricants a composition comprising at least one compound (I) wherein R_1, R_2, R_3, X and Y are as defined.

The following Examples illustrate the invention:

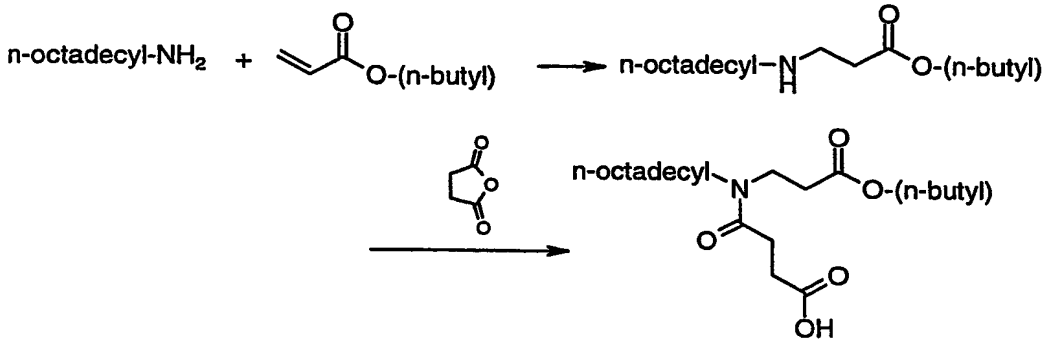
Examples

Temperatures are given in °C; h: hour(s); min.: minutes; m.p.: melting point; drying *in vacuo* (100°C, about. 0.05 mbar)

Synthesis Examples

Example 1

N-(2-n-Butoxycarbonylethyl)-N-(n-octadecyl)-succinic acid monoamide



1.1 14.1 g (0.110 mol) of acrylic acid n-butyl ester are added drop wise, at 60°C, over the course of 10 min., to 28.4 g (0.100 mol) of n-octadecylamine and the clear reaction mixture is stirred at 100° for 2 h; the cooled crude product is dissolved in 100 ml of

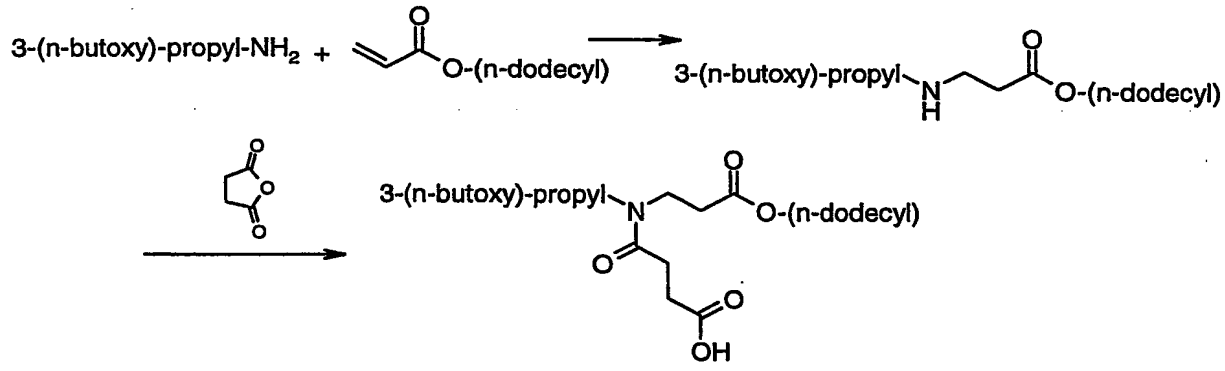
hexane, filtered and washed twice with 50 ml of water. The organic phase is concentrated by evaporation in a rotary evaporator, the residue is dried *in vacuo* and 40.0 g of a clear, colourless, medium-viscosity oil are obtained.

1.2 3.1 g (0.030 mol) of succinic anhydride are added to 11.9 g (0.030 mol) of the resulting intermediate product, and the clear reaction solution is stirred at 100° for 1 h. The cooled crude product is dissolved in 100 ml of hexane, filtered and washed twice with 40 ml of water. The hexane solution is concentrated by evaporation in a rotary evaporator and the residue is dried *in vacuo*. 13.7 g (92 % of theory) of a clear yellow oil, which crystallises on standing are obtained.

M.p.: 50-52°C; elemental analysis: 70.24 % C (calc. 69.98), 10.91 % H (calc. 11.14), 2.80 % N (calc. 2.81).

Example 2

N-(3-n-Butoxypropyl)-N-(2-n-dodecyloxycarbonylethyl)-succinic acid monoamide



2.1 26.7 g (0.10 mol) of acrylic acid n-dodecyl ester are added drop wise, over the course of 20 min., to 13.3 g (0.100 mol) of 3-n-butoxypropylamine and the reaction mixture is stirred at 100° for 2 h; the cooled crude product is dried *in vacuo* and 37.0 g of a clear, colourless, low-viscosity oil are obtained.

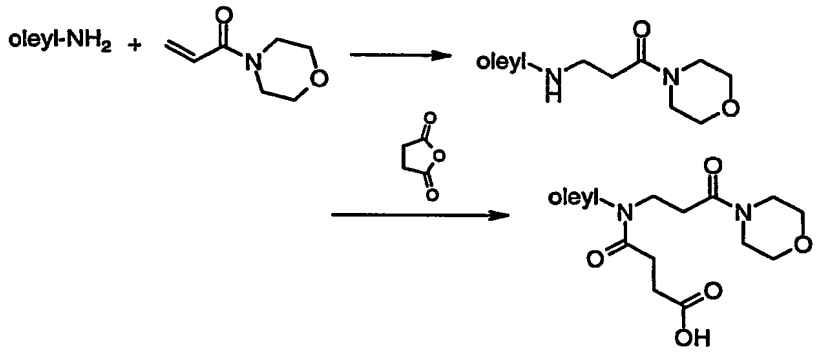
2.2 4.2 g (0.040 mol) of succinic anhydride are added to 14.9 g (0.040 mol) of the resulting intermediate product and the reaction mixture is stirred for 2 h; the cooled crude product is dissolved in 100 ml of hexane, filtered and washed three times with 30 ml of water. The hexane solution is concentrated by evaporation in a rotary evaporator, the remaining product is dried *in vacuo* and 18.0 g (95 % of theory) of a clear yellow oil of medium viscosity are obtained.

n_D^{20} 1.4670; elemental analysis: 66.85 % C (calc. 66.21), 10.54 % H (calc. 10.47), 2.72 % N (calc. 2.97).

n_D^{20} 1.4670; elemental analysis: 66.85 % C (calc. 66.21), 10.54 % H (calc. 10.47), 2.72 % N (calc. 2.97).

Example 3

N-(2-Morpholinocarbonylethyl)-N-(n-octadecyl)-succinic acid monoamide



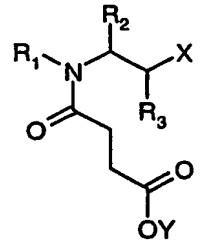
3.1 5.8 g (0.040 mol) of 4-acryloylmorpholine are added drop wise, at 60°, over the course of 10 min., to 10.7 g (0.040 mol) of oleylamine (Armeen®O), the reaction mixture is stirred at 100° for 1 h and the cooled crude product is dried *in vacuo*. 16.0 g of a clear, light-yellow, low-viscosity oil are obtained.

3.2 4.1 g (0.040 mol) of succinic anhydride are added to 16 g (0.040 mol) of the resulting intermediate product, the reaction solution is stirred at 100° for 1 h and the cooled crude product is shaken with 200 ml of hexane and 100 ml of brine (NaCl-saturated). The middle phase of the three phases is washed a further three times using 30 ml of brine each time and is dissolved in 100 ml of toluene, filtered and concentrated by evaporation in a rotary evaporator; the remaining product is dried *in vacuo*. 15.6 g (77 % of theory) of a clear yellow oil which becomes turbid on cooling are obtained.

Elemental analysis: 68.24 % C (calc. 68.47), 10.23 % H (calc. 10.30), 5.34 % N (calc. 5.51).

Example 4

The following compounds (I) can be prepared analogously to Examples 1-3:



(I),

R_2, R_3, Y: H

Table 1

Ex.	R_1	X	Yield [% of theory]	Appearance	Analysis [found (calculated)]
4.1	oleyl	-C(=O)-O-(n-butyl)	99	clear, light-yellow, medium-viscosity oil n_D^{20} 1.4761	70.79 % C (70.26) 10.58 % H (10.78) 2.82 % N (2.83)
4.2	oleyl	-C(=O)-O-methyl	93	clear, yellow, viscous oil n_D^{20} 1.4804	69.97 % C (70.08) 10.40 % H (10.44) 3.10 % N (3.10)
4.3	oleyl	-C(=O)-O$(CH_2)_2$-O-ethyl	96	clear, yellow, medium-viscosity oil n_D^{20} 1.4768	69.02 % C (68.07) 10.50 % H (10.44) 2.77 % N (2.74)
4.4	oleyl	-C(=O)-O-(isobutyl)	77	clear, yellow, medium-viscosity oil n_D^{20} 1.4748	70.04 % C (70.26) 10.76 % H (10.78) 2.80 % N (2.83)
4.5	oleyl	-C(=O)-O-ethyl	69	clear, yellow, medium-viscosity oil n_D^{20} 1.4769	68.85 % C (69.34) 10.50 % H (10.56) 2.97 % N (2.99)
4.6	isotridecyl	-C(=O)-O-(n-butyl)	90	clear, light-yellow, viscous oil n_D^{20} 1.4711	67.56 % C (67.41) 10.59 % H (10.61) 3.35 % N (3.28)
4.7	isotridecyl	-C(=O)-O-(isobutyl)	87	clear, light-yellow, viscous oil n_D^{20} 1.4709	67.63 % C (67.41) 10.66 % H (10.61) 3.36 % N (3.28)
4.8	n-butyl	-C(=O)-O-oleyl	95	clear, yellow, medium-viscosity oil n_D^{20} 1.4752	70.80 % C (70.26) 10.79 % H (10.78) 2.56 % N (2.83)
4.9	n-dodecyl	-C(=O)-O-ethyl	82	clear, light-yellow, medium-viscosity oil n_D^{20} 1.4706	65.78 % C (65.42) 10.39 % H (10.20) 3.72 % N (3.63)
4.10	oleyl	Nitrile	87	clear, light-yellow, viscous oil n_D^{20} 1.4783	72.13 % C (71.39) 10.73 % H (10.54) 6.23 % N (6.66)
4.11	n-octyl	-C(=O)-O-oleyl	97	clear, yellow, viscous oil n_D^{20} 1.4745	72.04 % C (71.69) 11.05 % H (11.30) 2.41 % N (2.53)
4.12	oleyl	-C(=O)-O-$(CH_2)_2$OH	59	clear, light-yellow, viscous oil n_D^{20} 1.4855	66.92 % C (67.05) 9.99 % H (10.21) 2.86 % N (2.90)
4.13	oleyl	-C(=O)-NH_2	82	clear, yellow, viscous oil n_D^{20} 1.4745	68.10 % C (68.45) 10.44 % H (10.57) 6.26 % N (6.39)

Table 1 (ctd.)

Ex.	R_1	X	Yield [% of theory]	Appearance	Analysis [found (calculated)]
4.14	coconut oil amine (C_{12} mix)	-C(=O)-O-(n-butyl)	87	clear, light-yellow, viscous oil n_D^{20} 1.4701	67.68 % C (67.73) 10.50 % H (10.18) 3.31 % N (3.29)
4.15	n-butoxy-propyl	-C(=O)-O-oleyl	97	clear, yellow, medium-viscosity oil n_D^{20} 1.4744	69.75 % C (69.40) 10.66 % H (10.74) 2.40 % N (2.53)
4.16	C_8-C_{10}alkyl-O$(CH_2)_3$	-C(=O)-O-(n-butyl)	95	clear, yellow, medium-viscosity oil n_D^{20} 1.4680	63.72 % C 10.00 % H 3.19 % N
4.17	isodecyl-O$(CH_2)_3$	-C(=O)-O-(n-butyl)	96	clear, yellow, medium-viscosity oil n_D^{20} 1.4699	64.75 % C (64.98) 10.11 % H (10.22) 3.14 % N (3.16)
4.18	2-ethylhexyl	-C(=O)-O-(n-dodecyl)	96	clear, light-yellow, medium-viscosity oil n_D^{20} 1,4684	69.60 % C (69.04) 10.69 % H (10.94) 2.67 % N (2.98)
4.19	methoxy-propyl	-C(=O)-O-(n-dodecyl)	98	wax-like solid	64.99 % C (64.31) 10.14 % H (10.09) 2.91 % N (3.26)
4.20	C_8-C_{10}alk-O$(CH_2)_3$	nitrile	97	clear, yellow, medium-viscosity oil n_D^{20} 1.4731	63.62 % C (64.38) 9.61 % H (9.67) 7.69 % N (7.90)
4.21	benzyl	-C(=O)-O-(n-dodecyl)	97	wax-like solid	70.02 % C (69.77) 8.99 % H (9.23) 3.10 % N (3.13)
4.22	oleyl	-C(=O)-O-cyclohexyl	98	clear, yellow, medium-viscosity oil n_D^{20} 1,4864	71,35% C (71,36) 10,12% H (10.62) 2,72% N (2,68)
4.23	oleyl	-C(=O)-O-$(CH_2)_2$-O-phenyl	98	clear, yellow, medium-viscosity oil n_D^{20} 1,5025	71,20% C (70,81) 9,57% H (9,54) 2,43% N (2,50)
4.24	oleyl	-C(=O)-O-$(CH_2)_2$-O-methyl	94	clear, yellow, medium-viscosity oil n_D^{20} 1,4791	67,82% C (67.43) 10,26% H (10.51) 2,85% N (2,81)
4.25	oleyl	-C(=O)-O-$(CH_2)_2$-O-$(CH_2)_2$-O-ethyl	94	clear, yellow, medium-viscosity oil n_D^{20} 1,4771	66.55% C (67.54) 10,55% H (10.34) 2,51% N (2,52)
4.26	benzyl	-C(=O)-O-dodecyl	98	yellowish, wax-like solid	

Table 1 (ctd.)

4.27	2-ethylhexyl	-C(=O)-O-dodecyl	96	clear, yellow, medium-viscosity oil n_D^{20} 1,4684	69,60% C (69,04) 10,69% H (10,94) 2.67% N (2,98)
4.28	n-butyl	-C(=O)-O-i-decyl	91	clear, yellow, medium-viscosity oil n_D^{20} 1,4708	65,24% C (65,42) 10,20% H (10,20) 3,43% N (3,63)
4.29	methyl-O-$(CH_2)_3$	-C(=O)-O-dodecyl	99	yellowish, wax-like solid	64,99% C (64,31) 10,14% H (10,09) 2,91% N (3,26)
4.30	ethyl-O-$(CH_2)_3$	-C(=O)-O-dodecyl	95	clear, yellow, medium-viscosity oil n_D^{20} 1,4681	65.25% C (64.98) 10,28% H (10,22) 2,85% N (3,16)
4.31	ethyl-O-$(CH_2)_3$	-C(=O)-O-oleyl	95	clear, yellowish, medium-viscosity oil n_D^{20} 1,4755	69,09% C (68,53) 10,76% H (10,54) 2,57% N (2.66)
4.32	i-propyl-O-$(CH_2)_3$	-C(=O)-O-oleyl	96	clear, yellow, medium-viscosity oil n_D^{20} 1,4741	68,19% C (68,98) 10,09% H (10,64) 2,55% N (2,59)
4.33	i-decyl-O-$(CH_2)_3$	-C(=O)-O-n-butyl	96	clear, yellow, medium-viscosity oil n_D^{20} 1,4699	64,75% C (64,98) 10,11% H (10,22) 3,14% N (3,16)
4.34	i-decyl-O-$(CH_2)_3$	-C(=O)-O-i-butyl	95	clear, yellow, medium-viscosity oil n_D^{20} 1,4696	64,87% C (64,98) 10,38% H (10,22) 3,25% N (3,16)
4.35	octadecyl-O-$(CH_2)_3$	-C(=O)-O-i-butyl	96	clear, yellow, medium-viscosity oil n_D^{20} 1,4682	66,76% C (67,30) 10,72% H (10,69) 2,91% N (2.80)
4.36	oleyl	-C(=O)-N$(CH_2)_5$	94	clear, yellow, medium-viscosity oil n_D^{20} 1,4985	70,76% C (71,10) 10,98% H (10,74) 5,24% N (5,53)

Application Examples

Example 5

Testing of corrosion protection properties (of active ingredient-containing industrial, circulating, turbine and hydraulic oils) according to ASTM D 665 (= DIN 51585)

300 ml of formulated oil are mixed with 30 ml of synthetic seawater by stirring for 24 h at 60° whilst simultaneously immersing a steel round-section bar.

After being in contact for that period, the steel bar is subjected to a visual corrosion test. Each test is carried out in duplicate. The base formulation is based on a mineral oil of speci-

fication SN VG46, which has been stabilised using antioxidants and metal deactivators. The active ingredients under test are used in a concentration of 0.1 mmol/kg (corresponding to 0.03 - 0.06 % by weight or 300 - 600 ppm). See Table 2 for results.

- 0: no corrosion
- 1: light corrosion (< 6 corrosion sites less than 1 mm in diameter)
- 2: moderate corrosion (< 5 % of the surface corroded)
- 3: heavy corrosion (> 5 % of the surface corroded)

Example 6

Testing for filterability in the presence of calcium ions

0.3 g of distilled water and 30 ppm of calcium in the form of a calcium detergent (6.9 % Ca) are added to 300 g of the test formulation. The mixture is homogenised in a four-blade mixer at the maximum speed of rotation for 5 min.. The emulsion is stored for 96 hours at 70°C (+/- 2°C) and then for a further 48 hours at room temperature in the dark. If precipitation is observed, the test is stopped. Before filtration, the test mixture is homogenised again by shaking vigorously for one minute. It is then filtered through a 0.8 µ Millipore® filter (type AAWP 04700) using compressed air at 1 bar (+/- 0.05 bar) positive pressure. The times required to filter 50, 100, 200 and 300 ml of test mixture are measured. The filtration index FI is calculated, as the mean of two measurements, in accordance with the following formula (see AFNOR NFE 48-691):

$FI = (t_{300ml} - t_{200ml})/ 2 (t_{100ml} - t_{50ml})$

FI = 1 means that no resistance is built up at the filter.

FI < 2 is the requirement for passing the test.

In the event of the filter becoming blocked, a note is made after 60 min. of the volume of oil filtered up until then.

Table 2

Example	Degree of corrosion, according to ASTM D 665	Filterability in the presence of Ca^{2+} Filtration index FI
Base formulation	3 / 3	1.25
1	1 / 0	
2	0 / 0	1.20
3	0 / 0	1.25
4.1	0 / 0	1.13
4.2	0 / 0	1.11
4.3	0 / 0	1.05
4.4	1 / 1	1.05
4.5	0 / 1	1.1
4.6	1 / 0	
4.7	0 / 0	
4.8	0 / 0	1.05
4.9	0 / 0	
4.10	0 / 0	1.05
4.11	1 / 1	
4.12	1 / 1	
4.13	1 / 1	1.20
4.14	1 / 0	
4.15	0 / 0	